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Acquisition of a Philips XL30 FEG SEM for the Microscopy Lab at The University of North Carolina at Wilmington

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13. ABSTRACT *(Maximum 200 words)*

Funds were sought for the acquisition of a Philips XL30 FEG scanning electron microscope (SEM) with a backscattered electron detector, an integrated EDAX non-dispersive X-ray microanalysis system, and a Cressington 208 HR turbo sputter coater. The high-resolution XL30 and its support equipment would replace our previous ISI SX40 SEM, which had extremely limited capabilities. The funds ($220,000) from the Office of Naval Research constituted 58.8% of the final cost of the instrumentation and were crucial in enabling the investigators to subsequently acquire the balance of the funds from the National Science Foundation ($41,842 – 11.2%) and the North Carolina Biotechnology Center ($112,218 – 30%). The instrument has been successfully installed and the principal investigator and microscopy technician have been trained in its use. The instruments have met or exceeded our expectations and every day more and more students and faculty are taking advantage of their potential. The impact of these instruments to the UNC Wilmington Microscopy laboratory and the University as a whole will be immeasurable, as it will greatly enhance our research capabilities.

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Final Report

Grant #: N0014-99-1-0690

Principal Investigator: Richard M. Dillaman, Ph.D.

Institution: University of North Carolina at Wilmington

Grant title: Acquisition of a Philips XL30 FEG SEM for the Microscopy Laboratory At the University of North Carolina at Wilmington

Award Period: 30/04/99-30/04/00

Objective: Funds were sought for the acquisition of a Philips XL30 FEG scanning electron microscope (SEM) with a backscattered electron detector, an integrated EDAX non-dispersive X-ray microanalysis system, and a Cressington 208 HR turbo sputter coater. The high-resolution XL30 and its support equipment would replace our present ISI SX40 SEM, which has extremely limited capabilities.

Approach: Funds ($220,000) from the Office of Naval Research constituted 58.8% of the final cost of the instrumentation and were crucial in enabling the investigators to subsequently acquire the balance of the funds from the National Science Foundation ($41,842 – 11.2%) and the North Carolina Biotechnology Center ($112,218 – 30%).

Accomplishments: The specific items of equipment acquired from FEI Company were:

Philips XL30S FEG without stage	$	279,922
Mains matching transformer for SEM	$	1,672
50 X 50 mm 4-axis motorized stage for XL30	$	31,757
Infrared inspection camera for WDS port	$	5,660
Low kV solid-state BS detector for S FEG	$	3,406
Mounting & preamplifier for backscatter detector	$	7,232
Frame store extension to eight images	$	3,338
Pentium PC for XL NT microscope	$	8,332
EDX completion kit	$	2,096
Video hardcopy unit	$	1,717
EDX digital controller	$	35,671
EDX multi-element mapping	$	6,812
EDAX SUTW detector for XL30	$	21,242
Water chiller for Philips XL SEM	$	4,600
Uninterrupted Power Supply – Profile Model 8	$	7,500
Total	$	420,957
Discount	$	71,818
Net Total	$	**349,139**

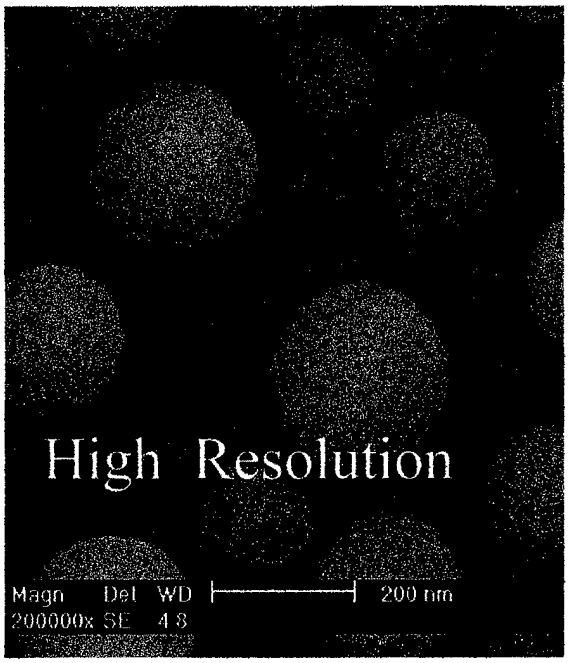
From Cressington Scientific Instruments a high resolution sputter coater was purchased:

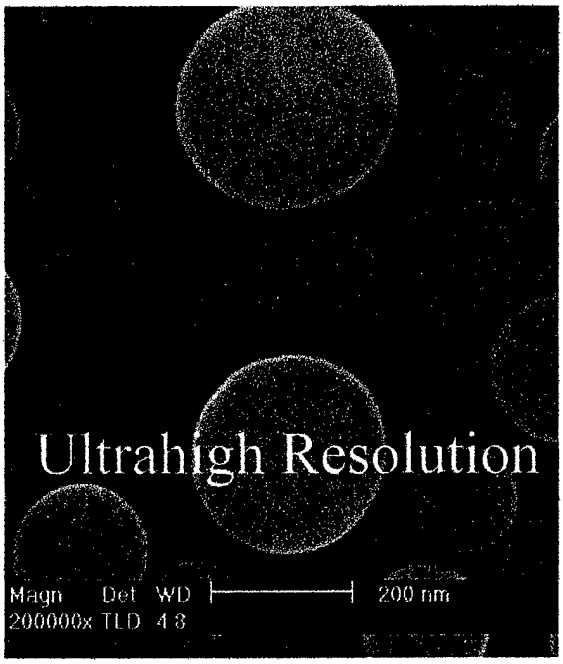
Cressington 208HR Turbo Sputter Coater	$	**24,921**
Total Equipment Costs	$	**374,060**

Changes from original equipment specifications**:

The equipment purchased varied slightly, but significantly, from that originally specified in the original grant proposal; and those two variations were:

1. Instead of purchasing an XL30 field emission scanning electron microscope (XL30 FEG SEM) as indicated, we purchased a XL30S FEG SEM. The difference between the two models is that the latter, in addition to standard secondary electron detector and solid state back-scattered electron detector, has an in-the-lens secondary electron detector. To use the terminology of the developers of this technology, while the XL30 is a ***high-resolution*** instrument, the XL30S is an ***ultra-high resolution*** instrument. The in-the-lens detector permits short working distances with high secondary electron detection. When this is coupled with the high beam densities afforded by the field emission gun, the result is an instrument that can routinely achieve 2-3 nm resolution. A comparison of the following electron micrographs of a standard sample (tin spheres sputter-coated with gold-palladium with the Cressington 208HR) clearly demonstrates the additional resolution that can be achieved with the XL30S.





2. The second feature that differs from the original specifications is that instead of having an oil diffusion pump to achieve a vacuum in the 3.0×10^{-6} mbar range, a turbomolecular pump was substituted. The advantage of the turbomolecular pump is that it is a purely mechanical pump and therefore does not have the potential of contaminating the column and specimen chamber. Diffusion pumps, which achieve their vacuum by vaporizing and condensing oil, always contaminate the microscope to some extent. The elimination of this source of contamination is particularly relevant when one is doing non-dispersive X-ray microanalysis. The silicon-based diffusion pump oils are often a source of contamination.

*****It should be noted that both of these improvements over the original specifications was obtained with no increase over the original estimated cost.***

Details of instrument installation:

The microscope arrived at UNC Wilmington on December 2, 1999 and on December 15, 1999 engineers from FEI (the new name for an expanded Philips Electron Optics) came and performed a site survey. During that survey they were pleased with the nature of the site, but made recommendations on the required types of electrical service

and plumbing modifications that would have to be made to support the instrument. The removal of the old SEM from the site and the completion of renovations necessary for the new microscope were completed by the end of February. On February 23, 2000 FEI engineers conducted a final facilities check and on March 2, 2000 installation began.

I wish I could say that the installation went smoothly, but it didn't. Several problems were encountered during the installation, including a failure of the photomonitor, a jammed table support that transferred vibrations to the column and several other minor problems that plagued the engineers. Most importantly, however, was the fact that after these annoyances were taken care of the resolution was nowhere near specifications. Several additional engineers were called in and finally a Dutch engineer was sent over from the factory and brought with him new deflector plates for the immersion lens. The team of 4 engineers worked for a whole week and in the end had the instrument working up to specifications (perhaps even exceeding them). The microscope was turned over to the University on **May 5, 2000**, more than 10 weeks after installation began! I must add here that I was impressed by the tenacity shown by the FEI engineers. They never walked away from the problem; there was an engineer here continuously over the entire interval and they brought more and more resources to bear as the installation became more protracted. New equipment means new problems and they seemed to accept this and answered the challenge.

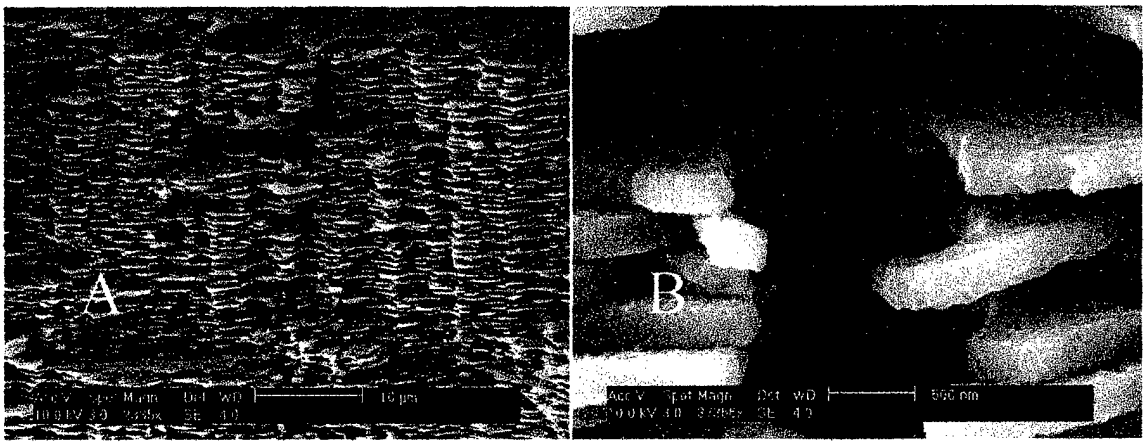
During the week following acceptance, the field engineer trained D. Mark Gay, the microscopy technician, and me on the routine operation of the microscope. I have to say that we were pleased with the ease of use of the microscope and were astounded by the wonderful resolution that we could achieve routinely. The plan was to familiarize ourselves with the microscope for several weeks and then to have further training by the Senior Field Application Engineer, Ove Thompson, who runs the applications lab in Portland, OR (where I had seen the XL30S demonstrated). That training occurred July 24-26, 2000 and during that training we received instruction on how to optimize the performance of the microscope, both from an instrumentation and sample preparation point of view. Here I must add that while learning the basic operation of the microscope we have also been testing the performance of the Cressington HR208, which was delivered after acceptance of the XL30S. It, likewise, is performing as expected.

Impact/Navy relevance:

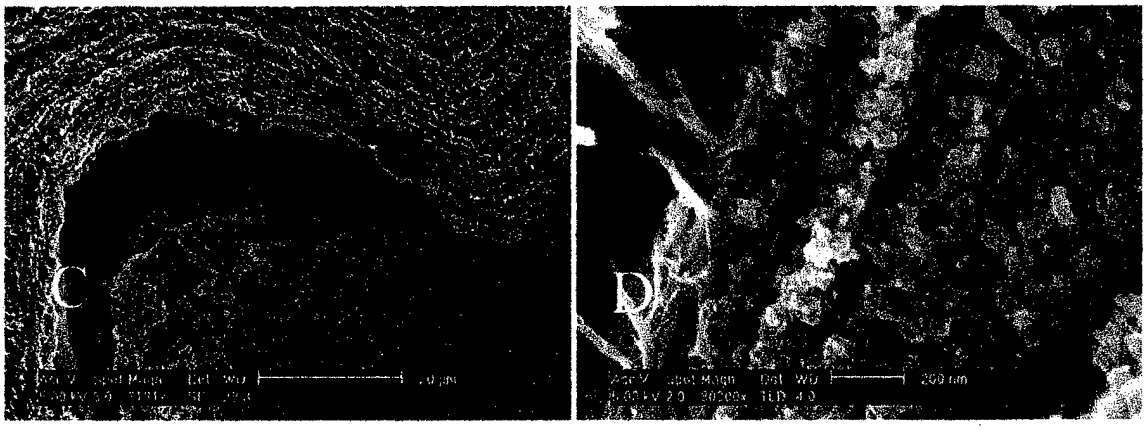
In summary, the Microscopy Laboratory at UNC Wilmington now has an ultrahigh resolution field emission scanning electron microscope, the Philips XL30S FEG, and is now able to support the programs and investigators that were described in our grant proposal to the Office of Naval Research, Science and Technology Section, Biomolecular and Biosystems Science and Technology Division. Although the microscope was not operational during the original period of the ONR grant, future investigators using this instrument will be requested to acknowledge ONR support of their research, particularly those individuals cited in the proposal.

Planned research efforts:

Currently investigators from both the Biology Department and Department of Earth Sciences are using the XL30S in their research programs. While as of yet no publications have resulted from the research using the images collected by the XL30S, attached below are just a few examples of the wide variety of materials being investigated.



The above images are from fossil material that is being examined for signs of dissolution over time. The image at the left (A) is that of a piece of shell showing the many individual crystals that determine the characteristic luster and mechanical properties of the shell. To the right (B) is a higher magnification of the individual crystals showing a large hole in the middle and the rough texture of the crystal surface.



Images C and D are from the cuticle of a molting crab. Figure C shows the multilayered chitin cuticle and the epithelial cells below (fractured and showing a hexagon shape) that are depositing the cuticle. Higher magnification of the surface of the epithelial cells (D) shows that the cuticle is polymerized on small projections of the cell membrane and fuse laterally to form a sheet.

Other sponsored science and technology:

Acquisition of a Philips XL30 FEG SEM for the Microscopy Laboratory at the University of North Carolina at Wilmington. National Science Foundation. $41,842, 4/01/99 – 3/31/00.

Acquisition of a Philips XL30 FEG SEM for the Microscopy Laboratory at the University of North Carolina at Wilmington. North Carolina Biotechnology Center. $112,218. 7/01/99 – 6/30/00.